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Indian Standard
SPECIFICATION FOR
ACID OIL

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR ACID OIL

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Indian Standard

SPECIFICATION FOR ACID OIL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 November 1986, after the draft finalized by the Oils and Oilseeds Sectional Committee had been approved by the Chemical Division Council and the Agricultural and Food Products Division Council.

0.2 Acid oil consists essentially of a mixture of fatty acids obtained by the treatment of soap stock with a mineral acid solution.

0.3 Acid oil is primarily produced by acidification of the soap stock obtained during refining of oils. The mineral acid in excess of the theoretical requirements is normally used along with heat and agitation to effect complete splitting of all soaps in the soap stock. This is followed by washing to remove all mineral acidity. The organic layer, settled and freed of separated water goes into storage or storage containers, usually of mild steel. Since acid oil is a bye-product of oil refining, the quantity may vary substantially, depending upon the crude oil processed for refining.

0.4 The acid oil, during storage and transport, picks up substantial quantities of iron, producing not only rusting of the containers but also deterioration of the fatty acids. These metals also catalyse oxidation of unsaturated acids producing a number of impurities which do not distil over as fatty acids. Acid oil is used for the manufacture of distilled fatty acids. Apart from this, the acid oil is also used for producing low grade soaps.

0.5 There is a separate Indian Standard (IS : 5638-1970*) which covers acid oil obtained from cottonseed oil or groundnut oil. The present standard does not cover the acid oil obtained from these sources.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Specification for acid oil (cottonseed and groundnut).

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for acid oil.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 11476-1985* shall apply.

3. REQUIREMENTS

3.1 The acid oil shall have an iodine value as agreed to between the purchaser and the supplier.

3.2 The acid oil shall conform to the requirements given in Table 1 when tested according to the methods referred to in col 4, 5 and 6 of Table 1.

TABLE 1 REQUIREMENTS FOR ACID OIL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL NO. OF		
			IS : 286-1978*	IS : 548 (Part 1)-1964†	Appedix A
(1)	(2)	(3)	(4)	(5)	(6)
i)	Total fatty matter (TFM), percent by mass, <i>Min</i>	95	15	—	—
ii)	Moisture and volatile matter content, percent by mass, <i>Max</i>	4.0	4	—	—
iii)	Free fatty acid, percent by mass, <i>Min</i>	50	—	7	—
iv)	Free mineral acid (as H_2SO_4), percent by mass, <i>Max</i>	0.02	—	—	A-2
v)	Unsaponifiable matter, percent by mass on TFM, <i>Max</i>	5.0	—	8	—
vi)	Oxidized fatty acid, percent by mass on TFM, <i>Max</i>	5.0	—	—	A-3
vii)	Iron, ppm, <i>Max</i>	500	—	—	A-4
viii)	Ash, percent by mass, <i>Max</i>	0.25	—	—	A-5

*Methods of sampling and test for soaps (*second revision*).

†Methods of sampling and test for oils and fats: Part 1 Sampling, physical and chemical test (*revised*).

*Glossary of terms relating to oils and fats.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in suitable containers or transported through tankers or as agreed to between the purchaser and the supplier.

4.2 Marking — The material, if packed in containers, shall be securely closed and marked with the following information:

- a) Name of the material;
- b) Manufacturer's name and recognized trade-mark, if any;
- c) Net mass of the material;
- d) Batch number or lot number in code or otherwise; and
- e) Month and year of manufacture.

4.2.1 Each container shall be legibly and indelibly marked with the information required under the *Standards of Weights and Measures (Package Commodities) Rules, 1977*.

4.2.2 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed under 3 of IS : 548 (Part 1)- 1964*.

*Methods of sampling and test for oils and fats: Part 1 Sampling, physical and chemical tests (*revised*).

A P P E N D I X A

(Table 1, Sl No. iv, vi, vii and viii)

TEST FOR FREE MINERAL ACID, OXIDIZED FATTY ACID, IRON AND ASH CONTENT

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF FREE MINERAL ACID

A-2.1 Apparatus

A-2.1.1 *Separating Funnel* — 250-ml capacity.

A-2.2 Reagents

A-2.2.1 *Methyl Orange Indicator* — Dissolve 0.1 g of methyl orange indicator in water and dilute to 100 ml.

A-2.2.2 *Standard Potassium Hydroxide Solution* — Aqueous, approximately N/50, accurately standardized, and stored in a bottle fitted with a guard tube of soda-lime to prevent access of carbon dioxide.

A-2.3 Procedure

A-2.3.1 Weigh, into the separating funnel, approximately 10 g of the material accurately to the nearest gram. Neutralize 100 ml of warm distilled water, using 0.1 ml of methyl orange indicator. Add the warm neutralized water to the material in the funnel, shake vigorously for one minute, and then allow to stand till the oily layer separates out. Run the aqueous layer into a flask, measure 50 ml of aqueous layer, add 0.1 ml of the methyl orange indicator, if necessary, and titrate with the standard potassium hydroxide solution.

A-2.4 Calculation

$$\text{Free mineral acid, percent by mass} = 56.1 \times \frac{VN}{M} \times 2$$

where

V = volume in ml of the standard potassium hydroxide solution used in the titration,

N = normality of the potassium hydroxide solution, and

M = mass in g of the material taken for the test.

*Specification for water for general laboratory use (*second revision*).

A-3. DETERMINATION OF OXIDIZED FATTY ACID

A-3.0 General

A-3.0.1 A known quantity of the material is saponified with alcoholic potash and the soap formed is treated with mineral acid to release the fatty acids. The petroleum ether insoluble but ethyl ether soluble matter, which is termed as oxidized fatty acid, is determined by evaporation of the ethyl extract of the residual aqueous layer left behind after the petroleum ether extraction.

A-3.1 Apparatus

A-3.1.1 *Stoppered Glass Cylinder* — 250-ml capacity.

A-3.1.2 *Separating Funnel* — 500-ml capacity.

A-3.1.3 *Flasks* — 250-ml capacity, flat bottom; and 300-ml capacity, conical.

A-3.2 Reagents

A-3.2.1 *Alcoholic Potassium Hydroxide Solution* — Dissolve 50 g of potassium hydroxide in 1 litre of 95 percent ethyl alcohol.

A-3.2.2 *Dilute Hydrochloric Acid* — 1:1 by volume.

A-3.2.3 *Methyl Orange Indicator* — Dissolve 0.1 g of methyl orange indicator in 100 ml of water.

A-3.2.4 *Petroleum Ether* — 60°C/80°C distillation range.

A-3.2.5 *Ethyl Ether*

A-3.3 Procedure

A-3.3.1 Weigh accurately 3 to 5 g of the fatty matter into a 300-ml conical flask. Add 50 ml of alcoholic potash, cover with an inverted funnel and heat on a water-bath to saponify. Agitate frequently and heat for at least 30 minutes or until saponification is complete.

A-3.3.2 Remove the watch-glass and continue heating on a water-bath with occasional agitation to evaporate the alcohol. To avoid oxidation, do not evaporate beyond a pasty mass. If necessary, add a small amount of water when most of the alcohol has evaporated.

A-3.3.3 Add 100 ml of distilled water and heat until the soap has completely dissolved. Wash the contents into glass-stoppered cylinder with hot distilled water, taking care not to exceed a total volume of 130 ml in the cylinder.

A-3.3.4 Add 3 to 5 drops of indicator and neutralize with hydrochloric acid to the pink methyl orange end point. Then add 1 ml of excess acid. Rotate the cylinder gently to mix the contents.

A-3.3.5 Cool to at least 35°C and add 125 ml of petroleum ether. The fatty acids need not have cleared completely before adding the ether. Stopper the cylinder, shake gently and allow to stand until the petroleum ether layer separates.

A-3.3.6 Siphon the petroleum ether layer into a 500-ml separating funnel, making sure that as little as possible of the insoluble matter which gathers at the ether-water interface is carried over into the separating funnel. If any appreciable amount of insoluble matter does siphon over into the separating funnel, it will usually settle to the bottom and shall be drained back into the extraction cylinder. Make at least 4 more similar extractions using 25 to 30 ml of petroleum ether, shaking the cylinder vigorously for 30 seconds with each extraction. Extractions shall be continued until the petroleum ether layer is practically colourless.

A-3.3.7 To the acid water remaining in the extraction cylinder, add 25 to 30 ml of ethyl ether, stopper, shake gently and allow to stand until the ether layer separates. Siphon the ethyl ether layer through a filter paper into a tared 250-ml flat bottom flask which has been dried and cooled in a desiccator. Make at least 4 more similar extractions using 25 to 30 ml of ethyl ether each time, and shaking the cylinder vigorously for 30 seconds with each extraction. The last ethyl ether extract shall be practically colourless.

A-3.3.8 Filter all extracts through the same filter paper and finally wash this filter paper thoroughly with ethyl ether to recover all the oxidized acids.

A-3.3.9 Evaporate the ethyl ether extracts on a water-bath under a gentle stream of clean dry air. Finally, dry the oxidized fatty acids in an air oven at $105 \pm 2^\circ\text{C}$ for 30 minutes. Cool in a desiccator to room temperature and weigh. Repeat until constant mass, that is, to within 0.1 percent between successive weighings, is obtained.

A-3.4 Reporting — Report the oxidized fatty acids as percentage of the material taken for the test.

A-4. DETERMINATION OF IRON CONTENT

A-4.0 Outline of the Method

A-4.0.1 The method is based on the isolation of metal from the soap by dissolving in hot water. The aqueous extract is treated with citric acid to sequester aluminium and then thioglycollic acid in ammoniacal solution is added and colour measured spectrophotometrically.

A-4.1 Apparatus

A-4.1.1 Spectrophotometer

A-4.2 Reagents

A-4.2.1 *Liquor Ammonia* — Relative density 0.9

A-4.2.2 *Dilute Sulphuric Acid* — 50 percent (v/v).

A-4.2.3 *Citric Acid (Aqueous Solution)* — 50 percent (v/v).

A-4.2.4 *Thioglycollic Acid (Aqueous Solution)*

A-4.2.5 *Standard Iron Solution* — Containing 10 μg of iron per ml prepared from ferric ammonium sulphate $[\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 12\text{H}_2\text{O}]$ in acid solution.

A-4.2.6 *Methyl Red Indicator* — 0.1 percent aqueous solution.

A-4.3 Procedure

A-4.3.1 *Isolation of Metals* — Weigh 50 g of the sample in a beaker and dissolve it with hot water. To this solution add 40 ml of concentrated hydrochloric acid with constant stirring and keep the beaker on steam bath until fatty acid layer separates. Add 20 g of paraffin wax to this solution while hot. Stir the solution at intervals and allow it to settle until phases are clear. Cool the mass to room temperature. Remove the wax cake with rod, rinse with water and add the rinsings to the aqueous phase. Evaporate the aqueous phase to about 60 ml by gently boiling. Add 100 ml of water and filter through paper washed previously with hydrochloric acid. Evaporate the filtrate and washings to about 60 ml. Cool and transfer the solution to a volumetric flask and make up the volume to 100 ml. Take aliquot portion of the solution for metal estimation.

A-4.3.2 *Determination of Iron* — Take 5 ml aliquot of the aqueous solution from the test solution in a 25-ml volumetric flask. To this add 4 ml citric acid solution and 0.02 ml methyl red indicator and liquor ammonia till the colour of the solution turns yellow. Then add 3 ml liquor ammonia in excess. Cool the solution and add 3 ml thioglycollic acid. Make up the volume to 25 ml and mix the solution thoroughly. Filter the solution through acid washed and dried filter paper. Measure absorbance of the clear solution at 540 nm in the spectrophotometer using water as reference. Prepare a calibration curve with standard iron solution and determine the iron content of the sample from it.

A-5. DETERMINATION OF ASH**A-5.1 Apparatus**

A-5.1.1 *Silica Dish* — 100-ml capacity.

A-5.2 Procedure

A-5.2.1 Heat the silica dish to redness, cool slightly, place in a desiccator, cool to room temperature and weigh. Repeat this operation until a constant mass is obtained. Add about 50 g of the material and heat gently on a burner until the sample ignites at the surface. Reduce the size of the flame until the heat is just enough to keep the sample burning.

A-5.2.2 When sufficient sample has burnt to permit addition of more, remove the heat source, allow to cool until the oil ceases burning, and then add a second weighed portion of the sample. A total of about 75 g, to be accurately weighed, should be used.

A-5.2.3 Continue heating the sample to a black char and transfer to an electric muffle furnace. Heat at 550 to 650°C for one hour.

A-5.2.4 Remove from the furnace, cool slightly, place in a desiccator and cool to room temperature. Weigh and repeat heating in the furnace to constant mass.

A-5.3 Calculation

A-5.3.1 Calculate the ash content as a percentage by mass of the material taken for the test.

(Continued from page 2)

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